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# PATENT SPECIFICATION

942,762

DRAWINGS ATTACHED.

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942,762



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## COMPLETE SPECIFICATION.

### A Method of Packing a Receptacle with Comminuted Material.

We, JOHN THOMPSON-KENNICOTT LIMITED, a Company registered under the laws of Great Britain, of Ettingshall, Wolverhampton, in the County of Stafford, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a method of packing a receptacle with comminuted material and particularly but not exclusively, to a method of packing a compartment of an electrodialysis apparatus with an ion exchange resin. The apparatus may, for example, be an electrodialysis apparatus as described in the Complete Specification of our co-pending Patent Application No. 14825/59 (Serial No. 893,051).

Some comminuted materials, for example ion exchange resins, are difficult to pack into a receptacle when wet because the particles become tacky and stick not only to one another but to anything else with which they come into contact. Such materials can be packed into the receptacle in the dry state but when they become wet, either due to absorption of water from the atmosphere or because they are wetted in use, they are subject to an increase in volume, which, in the case of an ion exchange resin, is of the order of 12%. In some cases this difficulty can be overcome by incompletely filling the receptacle by a calculated amount but this is not a satisfactory solution if the walls of the receptacle are constructed of a relatively flimsy material as, for example, the membranes of an electrodialysis apparatus compartment, when the pressure produced by the increase in volume of the comminuted material would deform the walls

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rather than cause the material to move and fill up the space that has been left in the receptacle.

It is an object of the present invention to provide an improved method of packing a receptacle with comminuted material in which the difficulties referred to above are materially reduced.

According to one aspect of the present invention a method of packing a receptacle with comminuted material comprises packing the material into the receptacle under pressure, supporting the walls of the receptacle to resist deformation thereof by the pressure applied and by any pressure produced by an increase in volume of the particles of the material and subsequently removing the support from the walls.

The material may be carried into the receptacle entrained in a stream of compressed air and the air allowed to escape. The air may be moist air and/or the method may be carried out in a humidity controlled atmosphere.

Advantageously when the material is such that it increases in volume on being wetted with water a part of the receptacle is left unpacked and the material is wetted to cause it to increase in volume to fill said receptacle before removing the support from the walls.

Alternatively the material is carried into the receptacle entrained in a stream of water and excess water is allowed to escape. The temperature of the material and of water trapped in interstices between particles of the material may then be lowered to cause the trapped water to freeze and bond the particles together.

If the material has been carried into the receptacle in a dry state, it may be wetted

with water and the temperature of the material and water may then be lowered to cause the water trapped in interstices between particles of the material to freeze and bond the particles together.

So that the invention may be clearly understood the application of the present method to the packing of a compartment of an electrodialysis apparatus will now be described by way of example, reference being made to the accompanying drawings in which:—

Figure 1 is a perspective view with parts broken away of a compartment of an electrodialysis apparatus;

Figure 2 is a section on the line II—II of Fig. 1;

Figures 3 and 4 each show diagrammatically means for feeding a resin into a compartment;

Figures 5 and 6 each show diagrammatically installations for feeding resin in the form of a slurry into a compartment; and

Figure 7 is a longitudinal section of a compartment prepared for filling with resin.

A simple electrodialysis apparatus comprises a unit of three compartments arranged so that the centre compartment is divided from the two outer compartments by permeable membranes. An electrode is placed in each of the outer compartments and the liquid to be processed is allowed to flow through the centre compartment. In a large electrodialysis apparatus there may be a large number of compartments disposed between the electrode compartments, each one being separated from the next adjoining compartment by a permeable membrane. One set of alternate compartments are termed concentrate compartments, i.e. the compartments into which ions migrate under the influence of the electric current passing between the electrodes, and the other set of alternate compartments are termed diluate compartments i.e. the compartments from which the ions migrate. Generally the volume of the concentrate compartments is made smaller than that of the diluate compartments, by making the distance between their bounding membranes smaller, because the flow of liquid through a concentrate compartment is made as small as possible from an economic point of view.

Each compartment whether diluate or concentrate, comprises an annular separator 1 of rectangular or square shape having on each side a permeable membrane 2 to form a closed central space 3. Passages 4 are formed in opposed separator ends (or sides) to permit the liquid to be dialysed to flow into and out of the central space 3 at a predetermined rate. Preferably means is provided to cause the liquid to flow over the whole surface of the membranes bounding the central space and for this purpose weirs

5 may be provided at the ends of the separator adjacent the passages.

The central space 3 may be packed with comminuted material 6 insoluble in the liquid being passed therethrough to act both as a support for the membranes 2 and to spread the liquid over the surface of the membranes. Ion exchange resins are particularly suitable for this purpose as they decrease the electrical resistance of the compartment if water containing a low concentration of salt is being desalted therein and also considerably decrease polarisation at the membrane surface. The ion exchange resins may be anion or cation resins or a mixture of both. When the central space 3 is packed with comminuted material means must be provided to prevent the material from passing out of the space 3 with the liquid being dialysed, such means may be the weirs 5 referred to above.

Gaskets 7 may be provided between the membranes 2 and the separator 1 to ensure that the liquid entering and leaving the central space 3 does so only through the passages 4 provided.

The separator may be provided with a sealable port (not shown) in one of its walls, other than the walls containing the liquid flow passages, to permit comminuted material to be introduced into the central space after that space has been sealed by the positioning of the membranes.

Separators of the type just described are described in greater detail in the Complete Specification of the Applicant's co-pending Application mentioned above.

The compartments may be filled according to the present method with the ion exchange resin in a dry state, a wet state or a dispersed state that is in the form of a thin slurry. Preferably the resin is packed into the compartment under sufficient pressure, for example at least 10 lb./sq. in., for the resin to resist further packing when the compartment is put into operation.

To feed the resin into the compartment in a dry state, the resin is first brought to the required degree of dryness by a stay in a humidity chamber. A predetermined quantity of the resin, which is 12% by volume less than that theoretically required to fill the compartment, is withdrawn from the chamber and passed to a feeder device. The feeder device comprises a chamber 8 having an outlet 9 communicating with the compartment and an inlet 10 through which compressed air at approximately 20 lb./sq. in. can be supplied. The compressed air, which is of the same humidity as the humidity chamber, forces the resin 11 into the compartment as will be described later.

To feed the resin into the compartment in the wet state, the resin is mixed with a quantity of water slightly in excess of that which

will be absorbed by the resin particles themselves to form a damp mass. A quantity 12 of the wet resin sufficient to fill the compartment is placed in a container 13 from 5 which it is forced as a "plug" through an outlet 14 and into the compartment by feeding water under pressure into the container 13 through an inlet 15.

To feed the resin into the compartment in 10 the form of a thin slurry, the resin is continuously fed from a source 22 into a receptacle 16 which is also supplied with water. The receptacle 16 is provided with a stirrer 17 which causes the resin to be uniformly 15 dispersed throughout the water. The resin slurry formed is fed via a pipeline 18 into the compartment by means of a pump 19 and the excess water escaping from the central space of the compartment through the 20 liquid inlet and outlet passage 4 is fed back to the receptacle 16 through a pipeline 20. Alternatively, water can be pumped into a mixing vessel 21 constantly supplied with resin from a source 22 so that the flow of 25 water through the vessel 21 causes the resin to be carried with the water into the compartment.

To fill the large number or "stack" of compartments forming the normal industrial 30 electrodialysis plant one of the following procedures is adopted.

A stack of compartments each comprising membrane, gasket, separator, gasket, membrane is first assembled with the central 35 space of each compartment left empty of resin. The membranes are then dried, as by passing air through the compartments, when they shrink and tighten like a drum skin. As the membranes do not immediately 40 assume their original dimension on being rewetted the resin is fed in the wet state, under a water pressure of at least 10 lb./sq. in. simultaneously into all of the compartments through the port provided in 45 the side wall of each separator.

If desired spacers may be inserted in the compartments to prevent the membranes sagging into contact with each other and so 50 that any tendency for the swelling resin to deform the membranes can be resisted by applying pressure at each end of the stack.

Another procedure which may be adopted 55 is to place a sheet 23 of polyethylene on a flat surface 24 for example a metal or plastic plate, and place a membrane 25, gasket 26, separator 27, gasket 28, membrane 29, in that order on top. A second sheet 30 of polyethylene is attached to a flat metal or 60 plastic plate 31 to prevent it sagging and is placed over the upper membrane 29. The polyethylene sheet 30 may be attached to the plate 31 by, for example applying water or a waxy or greasy substance e.g. petroleum 65 jelly, to a face of the plate before applying

the sheet to it or by generating electrostatic charges in the sheet and the plate by rubbing them. The polyethylene sheets 23 and 30 are provided to prevent the membranes 25 and 29 when wet from sticking to the plates 70 34 and 31 respectively. Pressure is then applied to the upper plate 31 either by means of a press or by clamping the upper and lower plates 24 and 31 together so that 75 the gaskets 26 and 28 are compressed thereby making the compartment water tight around its periphery. Resin is now fed into the compartment through a port (not shown) provided in the side wall of the separator. 80 The resin may be in the dry state, wet state or in the form of a thin slurry and is forced into the compartment at a pressure of 20—30 lb./sq. in. excess water of air escaping from the liquid flow passages.

While the separator 27 is held tightly 85 against the bottom plate 24, to prevent resin from passing between the lower membrane 25 and the separator 27, the pressure on the upper plate 31 is released and the plate 31 and polyethylene sheet 30 removed. A gasket, separator, gasket, membrane are then placed 90 on top of the first compartment to form the second compartment and the polyethylene 30 sheet and upper plate 31 replaced. Pressure is again applied to the upper plate and the second compartment is then filled in the 95 same manner as the first compartment. This procedure is repeated until the stack comprises the required number of compartments. 100

Lugs may be provided on the separator to enable pressure to be applied to it when the upper plate is removed.

It will be appreciated that if the compartments are being filled with dry resin, the resin has subsequently to be wetted and this is preferably left until after the next 105 following compartment has been filled.

In a further procedure the first stages are as just described except that the upper membrane is omitted and the resin is fed into the compartment through an aperture (not shown) formed substantially centrally of the upper metal plate 31 and polyethylene sheet 30. When the compartment is filled with 110 resin, the resin being wetted if the dry filling technique has been used, the temperature of the compartment is brought down to below the freezing point of the water and maintained there until a handleable mass of 120 resin particles held together by the formation of ice in the interstices between the particles is obtained. The pressure on the upper metal plate 31 is then released, the plate 31 and polyethylene sheet 30 removed 125 and the second membrane placed in position. The second and further compartments may now be built up on the first, each compartment being frozen after it has been filled and maintained in the frozen state 130

until the stack is complete and the electro-dialysis apparatus is erected.

If desired each compartment may be constructed separately and kept in cold storage until required to form part of a stack. In this case the pressure on the plate 31 is released and the plate removed but the polyethylene sheet 30 is allowed to remain in position to prevent evaporation of the water before it is frozen.

The polyethylene sheets 23 and 30 are provided to prevent the wet resin or membrane, as the case may be, from freezing on to the plates 24 and 31 respectively. It has been found that after freezing the plates can be easily parted from the polyethylene sheets and the latter can then be peeled off the frozen resin.

It has been found that the temperature of a resin filled compartment should not be reduced below about minus 10° C. otherwise the particles of resin may be ruptured due to the formation of crystals of frozen liquid within the particle itself. It will be understood that the liquid within the particle is a solution and will therefore have a lower freezing point than the substantially pure water added to the particles during the compartment filling process.

#### WHAT WE CLAIM IS:—

1. A method of packing a receptacle with comminuted material comprising packing the material into the receptacle under pressure, supporting the walls of the receptacle to resist deformation thereof by the pressure applied and by any pressure produced by an increase in volume of the particles of the material and subsequently removing the support from the walls.

2. The method according to Claim 1, wherein the material is carried into the receptacle entrained in a stream of compressed air and the air allowed to escape.

3. The method according to Claim 2, wherein the air is moist.

4. The method according to Claim 2 or 3, wherein the packing is effected in a humidity controlled atmosphere.

5. The method according to Claim 2, 3 or 4 wherein when the material is such that it increases in volume on being wetted with water a part of the receptacle is left unpacked and the material is wetted to cause it to increase in volume to fill said receptacle before removing the support from the walls.

6. The method according to Claim 1, wherein the material is carried into the receptacle entrained in a stream of water and excess water is allowed to escape.

7. The method according to Claim 6, wherein the temperature of the material and of water trapped in interstices between particles of the material is lowered to cause the trapped water to freeze and bond the particles together.

8. The method according to Claim 1 or Claim 2, wherein subsequent to its packing within the receptacle the material is wetted with water and the temperature of the material and water is lowered to cause the water trapped in interstices between particles of the material to freeze and bond the particles together.

9. A receptacle packed with comminuted material by the method of any one of the preceding claims.

10. A compartment of an electro-dialysis apparatus packed with a comminuted ion exchange resin by the method of any one of Claims 1 to 8.

11. A method of packing a receptacle with comminuted material substantially as herein described with reference to Figs. 1 and 2 and any one of Figs. 3 to 7 of the accompanying drawings.

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Fig. 1.

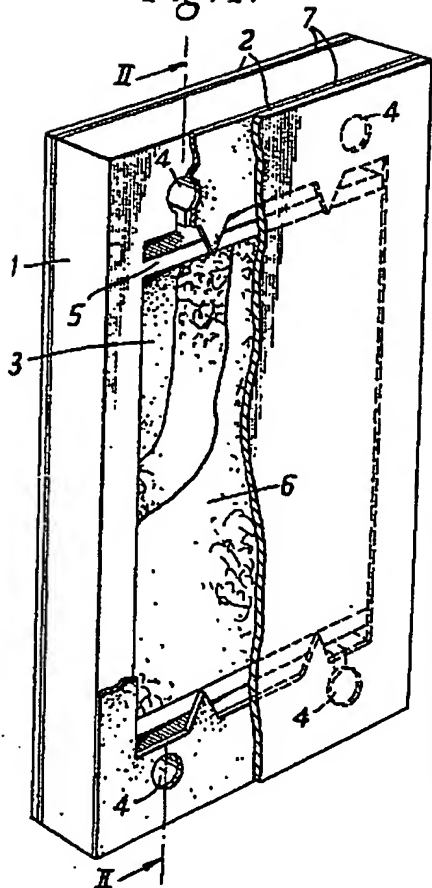


Fig. 2.

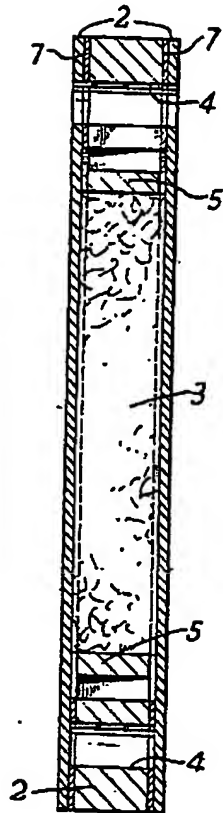


Fig. 2.

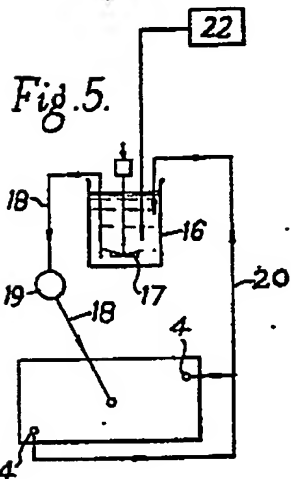
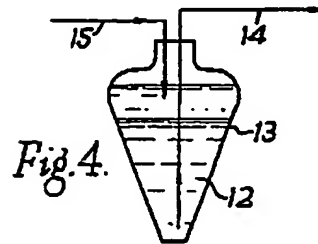
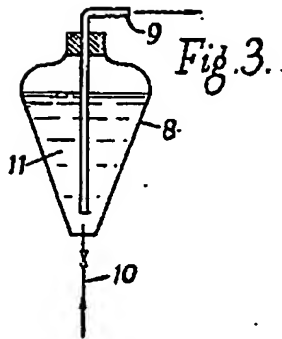
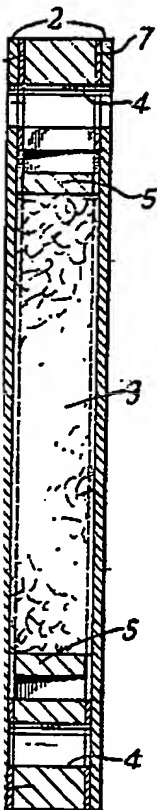


Fig. 6.

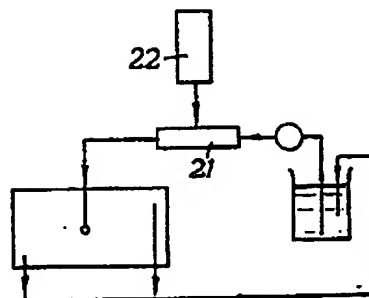


Fig. 7.

